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PHYSICAL METALLURGY DIVISION
INTERNAL REPORT PM-R-71-10

A STUDY OF THE PROPERTIES AND SINTERING BEHAVIOUR OF CERTAIN NICKEL POWDERS

Progress Report No. 4

by

C. F. Dixon and H. M. Skelly
Nuclear and Powder Metallurgy Section

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ABSTRACT

The sintering behaviour of four nickel powders and two series of blends of three of the powders was investigated. Compaction was carried out at three levels of pressure and sintering was done at 900°C (1650°F) for times of 1/2, 1 and 2 hr.

The mechanical properties, density and porosity of the sintered compacts were determined and the microstructure of representative specimens was examined.

Highest strength and greatest response to sintering conditions was shown by a fine ($\sim$ 6μ) dendritic powder. This powder also improved the sinterability of other powders, coarse (< 149 to > 105μ) and fine ($\sim$ 19μ) spheroidal, when blended with them.

Density and porosity were influenced more by compacting pressure than by sintering time. The porosity was mainly interconnected, even after sintering for 2 hr. Micro-examination confirmed the results of the density and porosity measurements.

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1. INTRODUCTION

The work described in this report is part of a study of the relationship between particle morphology and sintering behaviour for various nickel powders and blends. Earlier reports[1-3] have dealt with the properties of individual powders and blends and their behaviour under compaction. The present report gives the results of the first part of an investigation of the sintering behaviour of four powders and six blends.

2. TEST MATERIAL

The following nickel powders were investigated:

Powder Grade	Morphology	Particle Size, Microns
S	Spheroidal, pebbly	149 to <44
-100 + 140S	" "	<149 to >105
SF300	" "	~19
G-29	Loose, dendritic agglomerates	~6

Besides the four powders, the following blends were investigated: SF300 with 25%, 50% and 75% G-29, and -100 + 140S with the same percentages of G-29. The properties of the powders and blends have been described in earlier reports[1,3].

3. EXPERIMENTAL PROCEDURE

3.1 Compacting and Sintering

Compaction was carried out in a 100-ton (0.9 MN) hydraulic press. No lubricant was added to the powders, but the die walls were lubricated with stearic acid. Transverse rupture and tensile test pieces were prepared at three compacting pressures - 42.7, 64 and 96 ksi (295, 442 and 662 MN/m^2) for the former and 53.3, 66.7 and 80 ksi (368, 460

and 552 MN/m^2) for the latter. These compacting pressures were chosen after preliminary tests showed that they were the largest ranges that would give sound compacts for all powders and blends in the dies used.

The compacts were sintered in a hydrogen atmosphere at 900°C (1650°F) for times of 1/2, 1 and 2 hr for each compacting pressure.

3.2 Mechanical Testing

The strength of the sintered compacts was determined by carrying out transverse rupture and tensile tests on an Instron machine. The transverse rupture specimens, made to Metal Powder Industries Federation (MPIF) Standard 13-62, measured 1.25 in. (3.2 cm) by 0.5 in. (1.3 cm) by 0.25 in. (0.6 cm). The tensile specimens, made to MPIF Standard 10-63, were 3.5 in. (8.9 cm) long and 0.2 in. (0.5 cm) by 0.25 in. (0.6 cm) at the gauge section.

3.3 Density and Porosity

The density and porosity were determined for some of the sintered specimens, using the method described in MPIF Standard 8-50 in which the test sample is impregnated with oil under vacuum and weighed in air and water. The volume of oil taken up by the sample represents the interconnected porosity. The test samples used were halves of broken transverse rupture specimens.

3.4 Microexamination

Microexamination was carried out on representative sintered specimens taken from broken transverse rupture test pieces and impregnated and mounted in an epoxy resin prior to polishing.

4. RESULTS AND DISCUSSION

4.1 Mechanical Properties

The results of the transverse rupture tests for all powders and blends are listed in Table 1.

As inclusion of all possible plots of the mechanical test results would lead to an inordinately large number of graphs, it was decided to reproduce only a few, representative plots. Figures 1 and 2 are plots of transverse rupture strength against sintering time for powders and blends, all compacted at 64 ksi (442 MN/m^2). Both of these plots show that transverse rupture strength increases with increasing G-29 content. Also, the blends become more sensitive to sintering time with increasing G-29 content.

The effect of compacting pressure is shown in Figure 3, in which the transverse rupture strength of G-29 and -100 +140S is plotted against sintering time for three compacting pressures. Corresponding plots for the other powders and the blends fall in between the values for G-29 and -100 +140S. Figure 3 illustrates the comparative insensitivity of -100 +140S to sintering time for the three compacting pressures. Increasing the compacting pressure also has considerably less effect on -100 +140S than on G-29.

The tensile strength, yield strength and elongation results are listed in Tables 2, 3 and 4, respectively. Representative plots of the tensile values are given in Figures 4 to 6. The yield and elongation results were not plotted. The comments made above for the transverse rupture plots also apply to the plots of tensile strength. Thus, tensile strength of the blends increases with G-29 content and the latter powder is the most responsive to sintering time.

In general, transverse rupture and tensile strengths both increased with increasing compacting pressure for all powders and blends. The order

of increasing strength (transverse rupture and tensile) for the powders was -100 +140S, S, SF300 and G-29. The blends of G-29 with SF300 were generally stronger than those of G-29 with -100 +140S, and both blends increased in strength with increasing G-29 content.

4.2 Density and Porosity

The density and porosity of some representative specimens are listed in Table 5. Density is shown as g/cc and also as a percentage of the theoretical density (TD). The porosity as determined by the oil-impregnation method gives the interconnected porosity. Also shown in Table 5 is the total porosity (expressed as percentage of sample volume) calculated from the relationship:

total porosity = 100 - % theoretical density.

The density of -100 +140S is practically unaffected when the sintering time is changed from 1/2 hr to 2 hr, but increasing the compacting pressure from 42.7 to 96 ksi (295 MN/m^2 to 662 MN/m^2) results in a significant increase in sintered density.

Increasing the sintering time of the blend of -100 +140S with 50% G-29 produces a small increase in density, resulting from the high sintering activity of the G-29 powder. But a more significant increase in density occurs on increasing the compacting pressure. The higher densities of the blends compared with -100 +140S are due to the presence of G-29 in the former.

It is noteworthy that, in all cases, the porosity is mainly interconnected.

4.3 Microstructure

Some typical microstructures are shown in Figures 7 to 14. These were taken from the specimens whose densities and porosities are listed in Table 5. Comparison of Figures 7 and 8 and of Figures 9 and 10

illustrates how little the density of -100 +140S changes between sintering times of 1/2 and 2 hr. Comparison of Figures 7 and 9 and of Figures 8 and 10 illustrates the increase in density from a compacting pressure of 42.7 ksi (295 MN/m^2) to one of 96 ksi (662 MN/m^2). Increasing the sintering time from 1/2 to 2 hr results in large voids opening up within the powder particles -- presumably at the expense of smaller voids -- and this would appear to offset, to some extent, an increase in density resulting from movement of metal to close pores and reduce surface tension.

The above remarks also apply to Figures 11 to 14, photomicrographs of -100 +140S with 50% G-29, except that there does not appear to be as significant an increase in void size within the powder particles of -100 +140S on increasing sintering time from 1/2 to 2 hr; this would account for the slight increase in density of the blend on increasing the sintering time from 1/2 to 2 hr.

5. CONCLUSIONS

1. Of the four powders examined, highest sintered strength is shown by the fine (∿ 6μ) dendritic powder. The other three powders, in order of decreasing strength, are the fine (∿ 19μ) spheroidal, the mixed (149 to < 44μ) spheroidal and the coarse (< 149 to >105μ) spheroidal.

2. The sintered strength of the spheroidal particles, coarse (< 149 to > 105μ) or fine (∿19μ), increases with increasing blended additions of fine (∿ 6μ), dendritic particles.

3. The strength of the fine dendritic powder improves at a faster rate with increasing sintering time and compaction pressure than do the strengths of the coarse or fine spheroidal powders.

4. Blending the spheroidal powders with the dendritic powder increases their response to sintering time and compaction pressure.

5. Increased sintering time has little effect on the density of coarse spheroidal powder, but blending with fine dendritic powder results in an increase in density with sintering time.

6. After 2 hr of sintering, the porosity in compacts of coarse spheroidal powder and blends with the fine, dendritic powder, is mainly interconnected.

6. ACKNOWLEDGEMENT

Most of the laboratory work was performed by Mr. J. McGoey, Technical Assistant in this Section.

7. REFERENCES

1. H. M. Skelly and C. F. Dixon. "A Study of the Properties and Sintering Behaviour of Certain Nickel Powders, Progress Report No. 1", Physical Metallurgy Division Internal Report PM-R-68-9, Mines Branch, Department of Energy, Mines and Resources, Ottawa (April 23, 1968).

2. H. M. Skelly and C. F. Dixon. "A Study of the Properties and Sintering Behaviour of Certain Nickel Powders, Progress Report No. 2", Physical Metallurgy Division Internal Report PM-R-68-18, Mines Branch, Department of Energy, Mines and Resources, Ottawa (November 5, 1968).

3. H. M. Skelly and C. F. Dixon. "A Study of the Properties and Sintering Behaviour of Certain Nickel Powders, Progress Report No. 3", Physical Metallurgy Division Internal Report PM-R-71-6, Mines Branch, Department of Energy, Mines and Resources, Ottawa (April 30, 1971).

TABLE 1

Transverse Rupture Strength of Sintered Compacts

Sintering Temperature: 900°C (1650°F)

Compacting Pressure - ksi (MN/m^2)	Sintering Time (hr)	Transverse Rupture Strength (ksi)									
						-100 + 140S with:			SF 300 with:		
		S	-100 +140 S	SF300	G-29	25% G-29	50% G-29	75% G-29	25% G-29	50% G-29	75% G-29
42.7 (295)	½	1.60	1.64	4.32	24.65	6.87	15.55	21.08	8.38	15.76	19.47
	1	2.62	2.25	5.02	32.49	9.20	18.57	26.25	10.35	22.00	27.65
	2	3.96	2.69	6.07	40.77	9.90	22.40	32.30	13.39	23.10	30.06
64 (442)	½	8.66	3.07	7.56	32.47	9.50	21.80	28.60	12.84	20.40	27.99
	1	7.81	3.97	8.76	41.48	11.35	23.05	33.20	15.50	24.20	33.02
	2	8.02	5.35	10.36	51.65	13.59	30.40	39.25	19.55	32.60	39.55
96 (662)	½	15.26	5.44	12.43	43.85	15.90	26.60	36.50	21.60	29.10	38.40
	1	14.40	6.35	14.45	58.82	17.50	31.70	45.50	27.25	36.05	43.79
	2	16.48	8.96	19.73	67.49	19.75	39.30	54.30	33.70	40.20	54.58

Note: For the sake of clarity, the strength values in Tables 1 to 3 are given in ksi only. The approximate MN/m^2 equivalent can be obtained by multiplying the ksi figure by 6.9.

TABLE 2

Tensile Strength of Sintered Compacts

Sintering Temperature: 900°C (1650°F)

Compacting Pressure, ksi (MN/m^2)	Sintering Time (hr)	Ultimate Tensile Strength (ksi)									
						-100 + 140S with:			SF300 with:		
		S	-100 +140 S	SF300	G-29	25% G-29	50% G-29	75% G-29	25% G-29	50% G-29	75% G-29
53.3 (368)	½	1.0	0.9	2.73	18.10	4.85	7.40	15.66	6.05	11.20	13.33
	1	1.70	1.10	4.10	22.10	4.70	13.00	17.90	7.10	12.40	16.40
	2	2.37	1.63	4.90	24.85	6.32	13.70	21.60	9.43	13.72	21.32
66.7 (460)	½	1.34	1.18	4.22	22.30	5.45	11.50	15.80	7.08	12.80	17.65
	1	2.40	1.50	5.40	25.73	6.30	14.60	16.30	9.70	14.70	20.20
	2	2.80	2.00	6.40	31.55	7.13	17.85	20.20	11.78	19.17	25.22
80 (552)	½	2.33	1.54	4.35	24.40	5.30	13.80	17.55	9.66	14.70	19.50
	1	3.00	1.70	6.90	29.20	6.80	16.00	22.40	12.70	18.50	23.20
	2	4.33	2.35	9.02	34.70	8.84	17.60	28.85	14.43	22.28	28.90

TABLE 3

Yield Strength of Sintered Compacts

Sintering Temperature: 900°C (1650°F)

Compacting Pressure, ksi (MN/m^2)	Sintering Time (hr)	Yield Strength (0.2% offset) ksi									
		S	-100 +140 S	SF300	G-29	-100 +140S with:			SF300 with		
						25% G-29	50% G-29	25% G-29	25% G-29	50% G-29	75% G-29
53.3 (368)	½	0.91	Nil	2.40	13.30	Nil	Nil	12.94	5.96	9.13	11.60
	1	1.60	Nil	3.60	14.70	Nil	9.60	12.20	5.70	9.50	12.90
	2	1.84	1.30	3.57	14.65	5.03	8.98	12.63	6.08	9.36	12.97
66.7 (460)	½	Nil	Nil	3.30	15.90	Nil	10.50	14.10	6.38	10.40	13.60
	1	1.90	1.40	4.70	16.64	5.90	10.50	13.80	7.10	10.35	14.30
	2	2.04	1.63	4.59	17.10	5.57	10.18	12.98	7.34	11.27	14.39
80 (552)	½	1.98	1.44	3.70	17.25	Nil	12.00	14.90	8.00	11.70	15.90
	1	2.40	1.65	5.70	17.30	6.60	11.20	14.70	8.70	12.55	16.00
	2	2.52	1.86	5.68	18.20	6.47	10.13	14.80	8.56	12.73	16.13

TABLE 4

Elongation of Sintered Compacts

Sintering Temperature: 900°C (1650°F)

Compacting Pressure ksi (MN/m^2)	Sintering Time (hr)	Elongation (% on 1 in.)									
					-100 +140S with:				SF300 with:		
		S	-100 +140 S	SF300	G-29	25% G-29	50% G-29	75% G-29	25% G-29	50% G-29	75% G-29
53.3 (368)	½	1.0	6.0	2.0	4.0	*	1.0	3.5	1.0	2.0	2.0
	1	2.0	1.0	1.0	6.0	1.0	3.0	5.0	2.0	3.0	5.0
	2	1.0	1.0	2.0	8.0	2.0	4.0	7.0	6.0	5.0	8.0
66.7 (460)	½	Nil	*	*	5.0	1.0	2.0	3.0	1.0	2.0	5.0
	1	2.0	1.0	*	6.0	2.0	4.0	3.0	3.0	4.0	5.0
	2	2.0	1.5	*	12.0	*	4.0	*	5.0	7.0	10.0
80 (552)	½	Nil	2.0	1.0	7.0	1.0	2.0	3.0	1.0	*	5.0
	1	2.0	1.0	*	9.0	1.0	4.0	4.0	5.0	5.0	6.0
	2	*	2.0	4.0	14.0	*	4.0	11.0	8.0	*	*

*broke outside gauge marks.

TABLE 5

Density and Porosity of Specimens Sintered at 900°C (1650°F)

Specimen	Compacting Pressure ksi (MN/m^2)	Sintering Time (hr)	Density		Porosity %	
			g/cc	% TD	Inter-connected	Total
-100 +140S	42.7 (295)	½	5.36	60.2	34.8	39.8
"	"	2	5.35	60.1	35.6	39.9
	96 (662)	½	6.25	70.2	23.6	29.8
	"	2	6.27	70.4	24.6	29.6
-100 +140S with 50% G-29	42.7 (295)	½	6.47	72.7	24.7	27.3
"	"	2	6.79	76.3	22.7	23.7
"	96 (662)	½	7.26	81.6	15.3	18.4
"	"	2	7.36	82.7	14.7	17.3

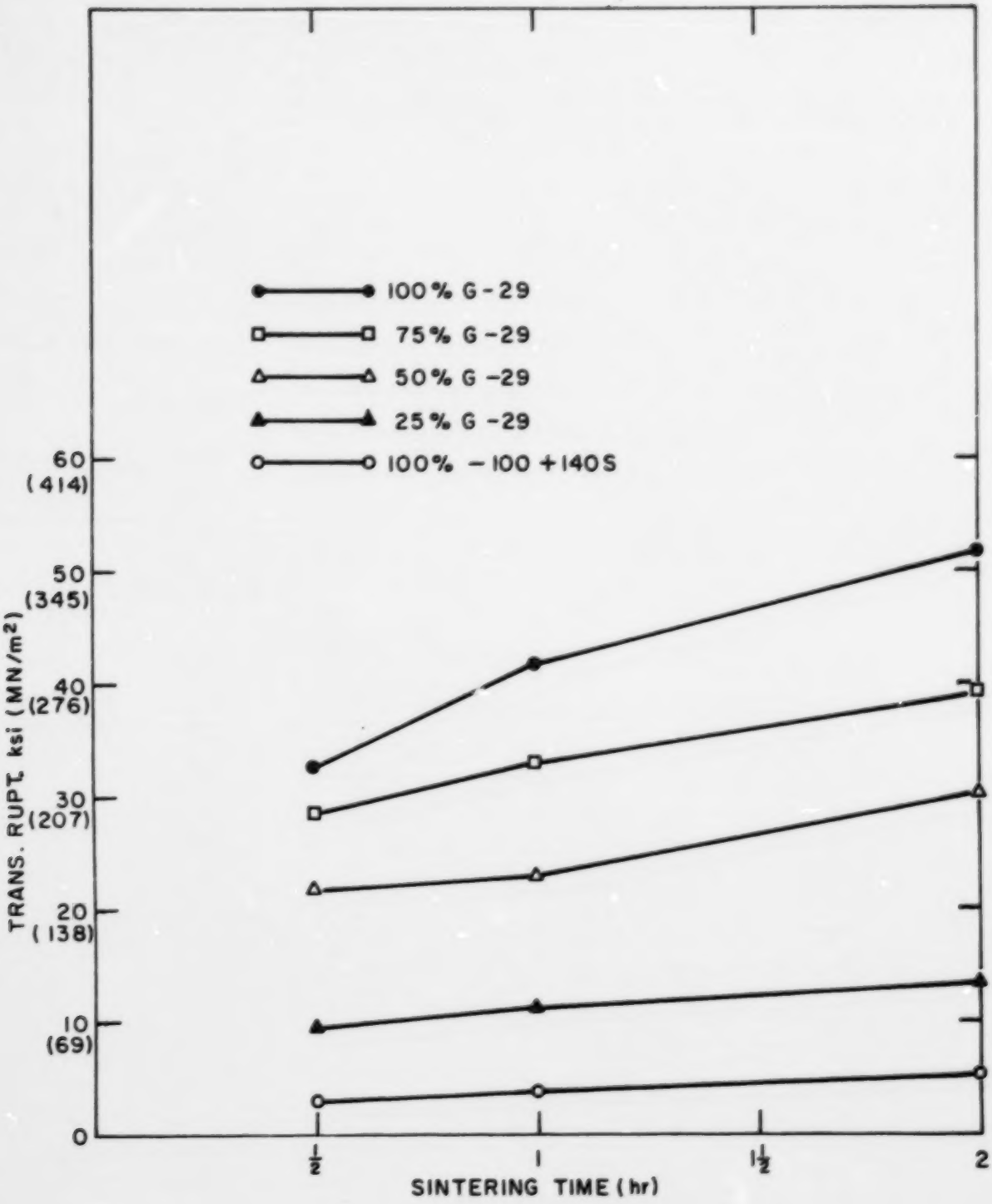


FIGURE 1. TRANS. RUPT. vs. SINTERING TIME FOR G-29, -100+140S AND BLENDS
COMPACTING PRESS: 64 ksi (442 MN/m^2) SINTERING TEMP: 900°C (1650°F)

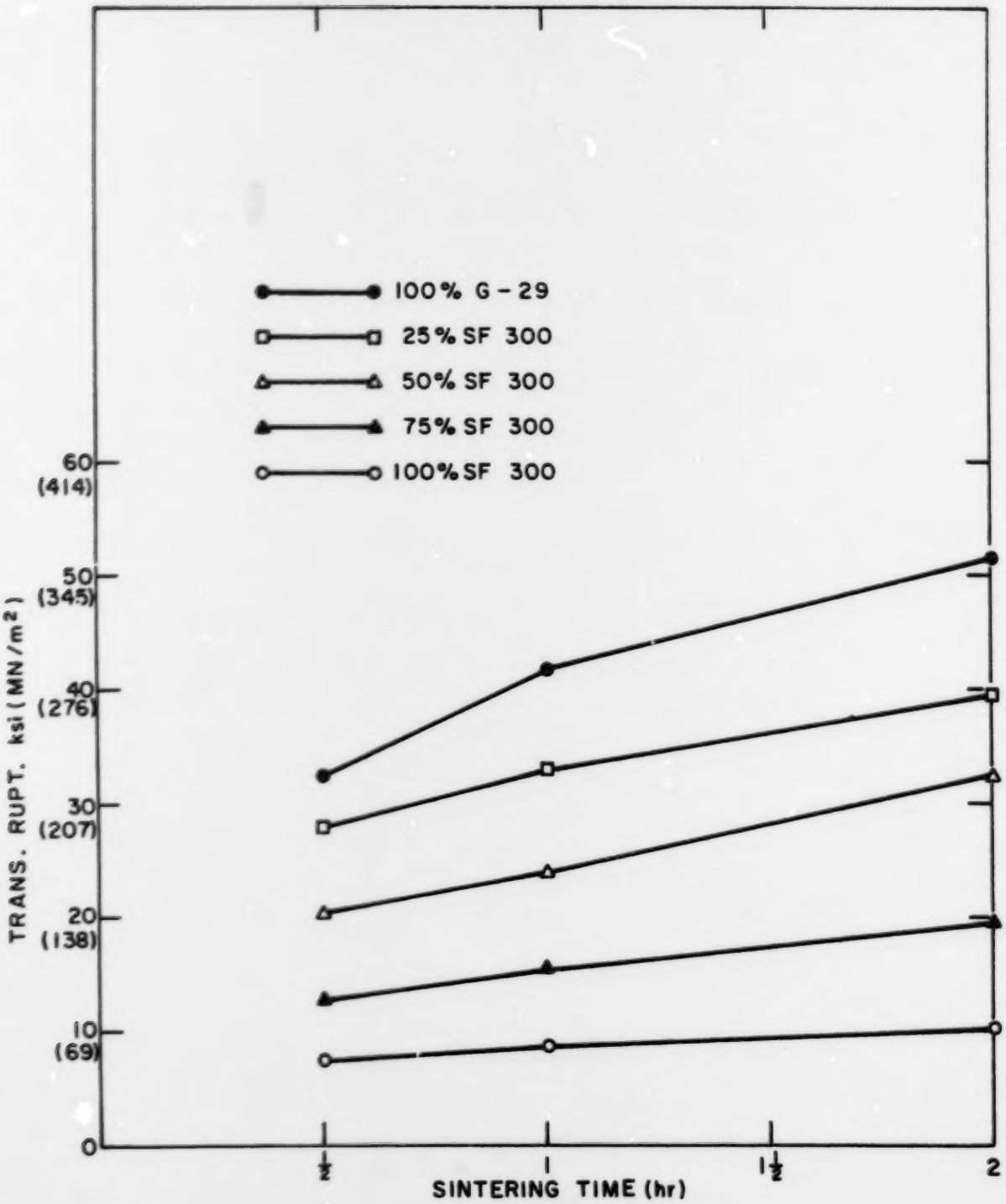


FIGURE 2. TRANS. RUPT. vs. SINTERING TIME FOR G-29, SF 300 AND BLENDS. COMPACTING PRESS: 64 ksi (442 MN/m^2) SINTERING TEMP: 900°C (1650°F).

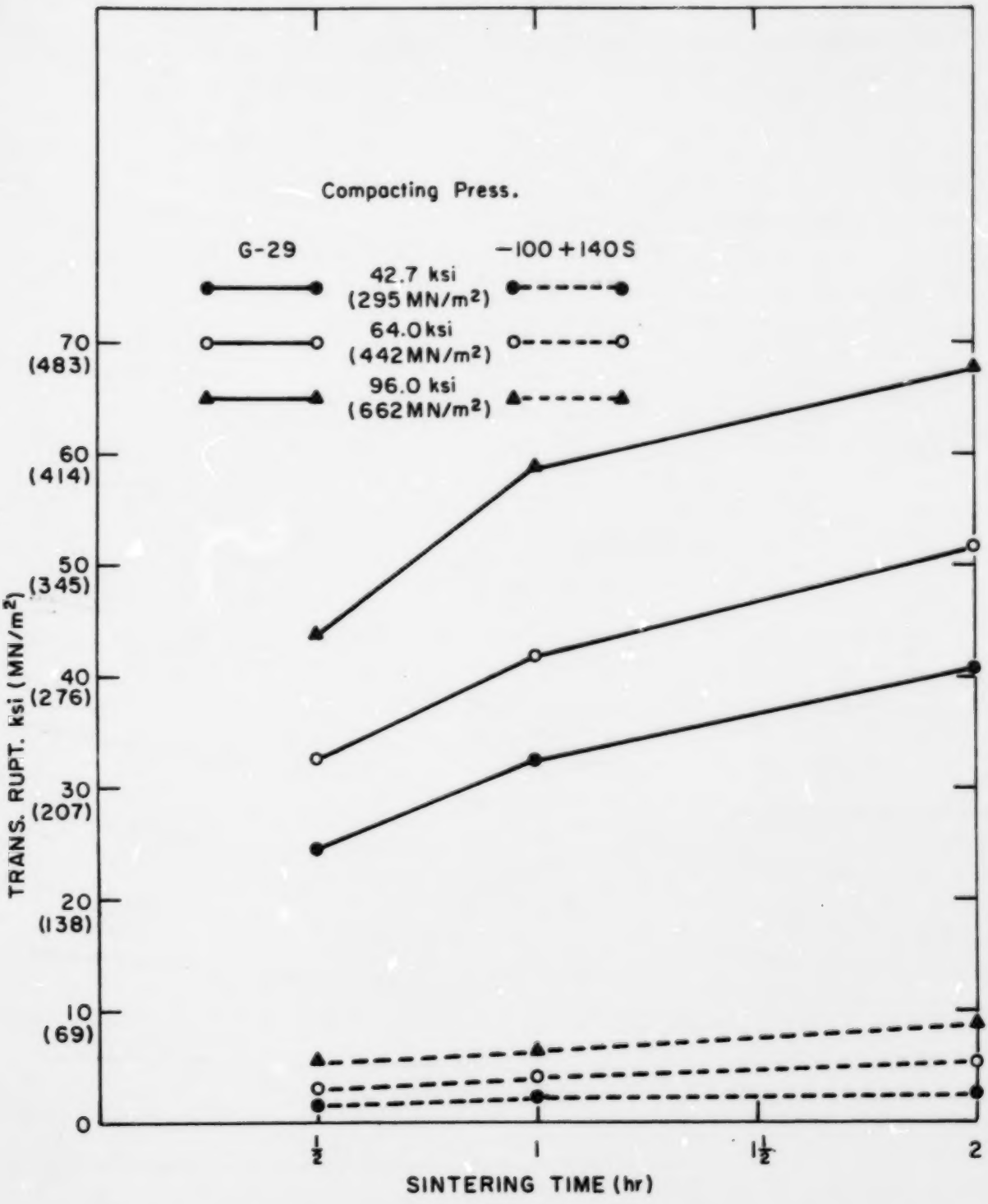


FIGURE 3. TRAN. RUPT. vs. SINTERING TIME FOR G-29 AND -100+140S POWDERS
SINTERING TEMP: 900°C (1650°F).

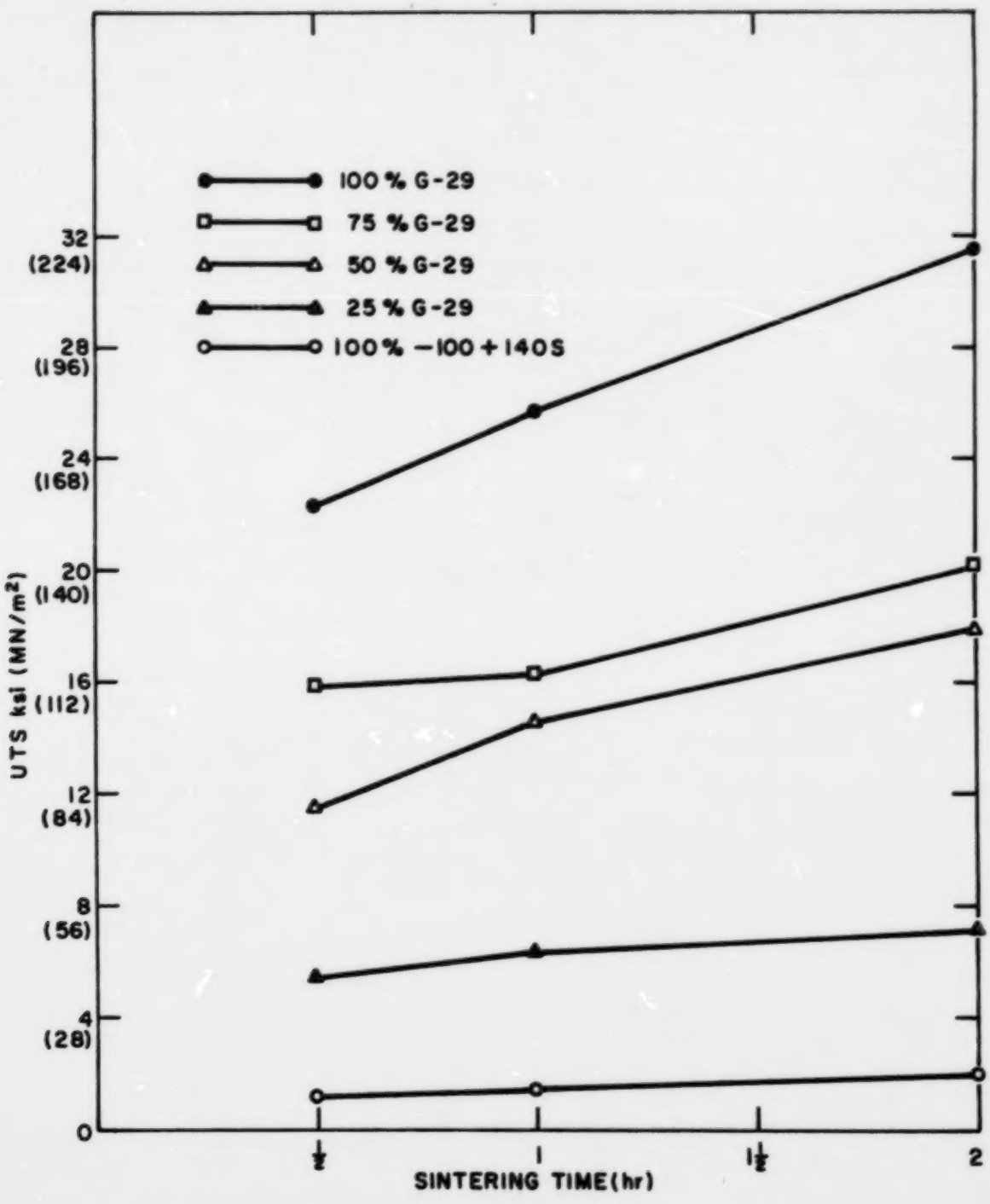


FIGURE 4. UTS vs SINTERING TIME FOR G-29, -100+140S AND BLENDS
COMPACTING PRESS: 66.7 ksi (460 MN/m^2) SINTERING TEMP: 900°C (1650°F)

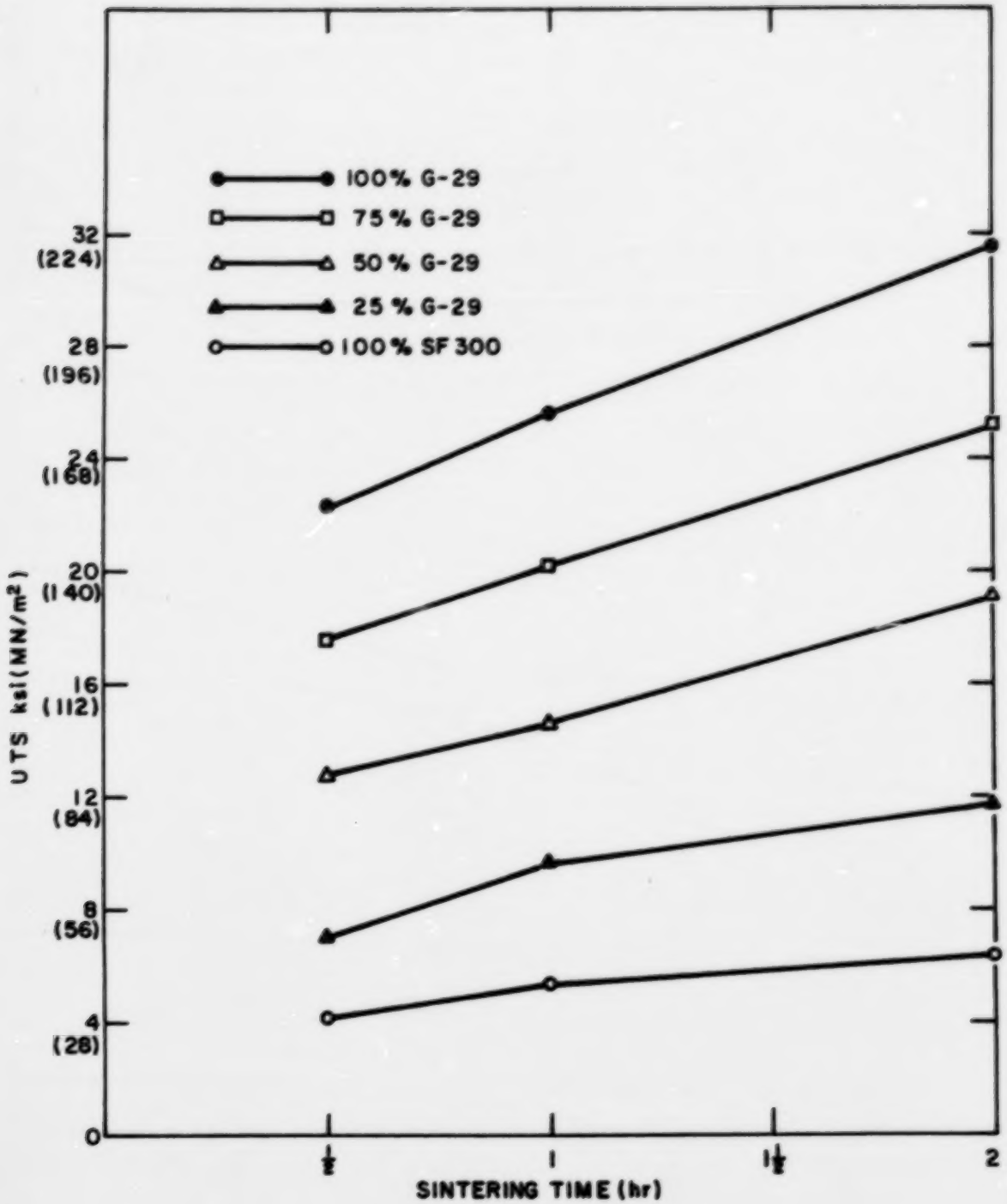


FIGURE 5. UTS vs SINTERING TIME FOR G-29, SF 300 AND BLENDS
COMPACTING PRESS: 66.7 ksi (460 MN/m^2) SINTERING TEMP. 900°C (1650°F).

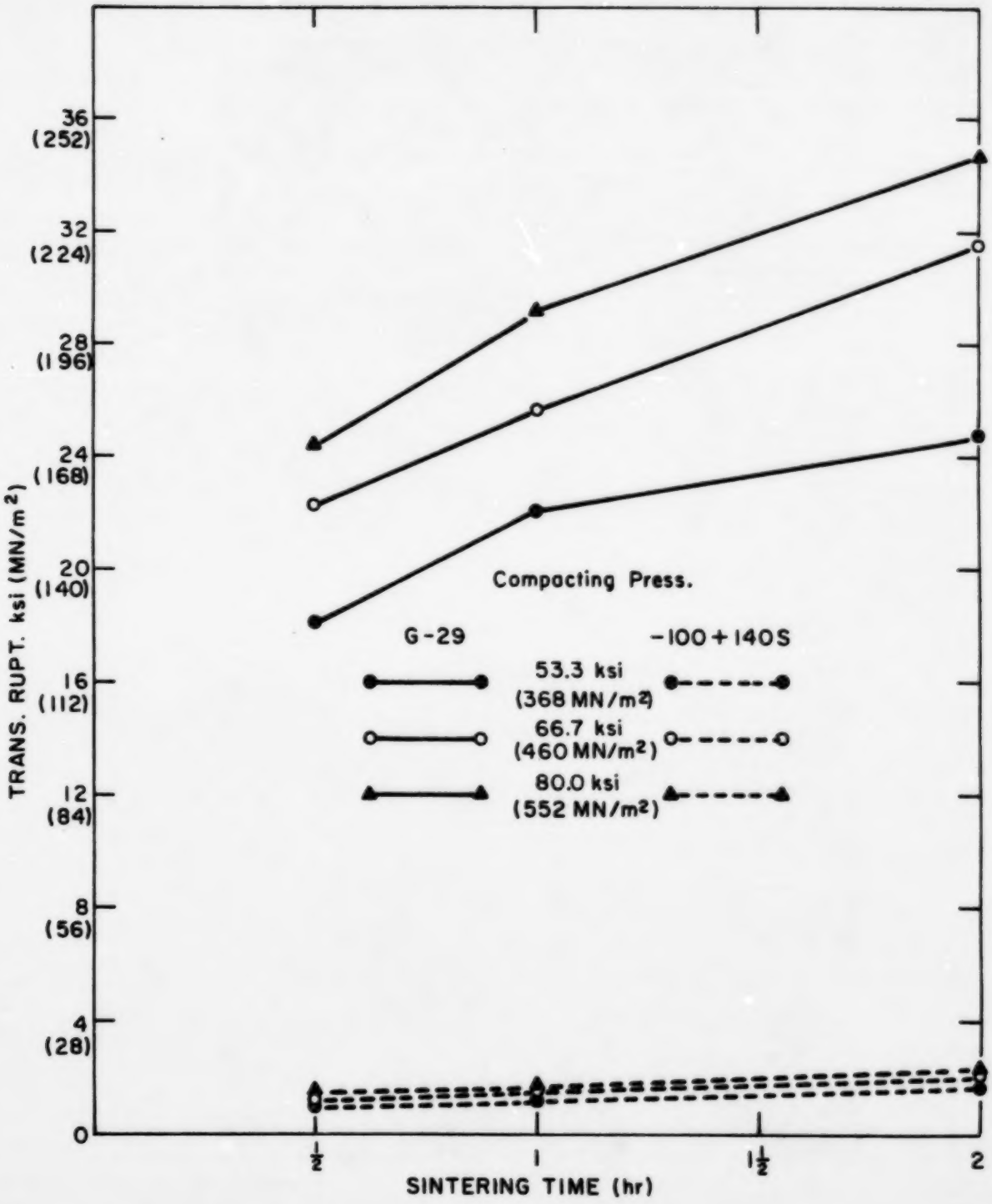
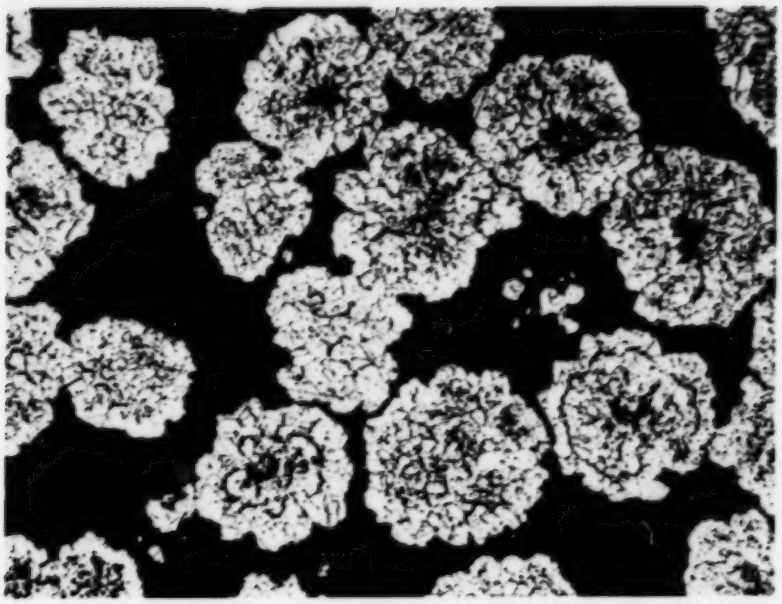


FIGURE 6. UTS vs SINTERING TIME FOR G-29, AND -100+140S POWDERS. SINTERING TEMP: 900°C (1650°F).



Etched X200

Figure 7. -100 +140S compacted at 42.7 ksi (295 MN/m^2) and sintered at 900°C (1650°F) for 1/2 hr.

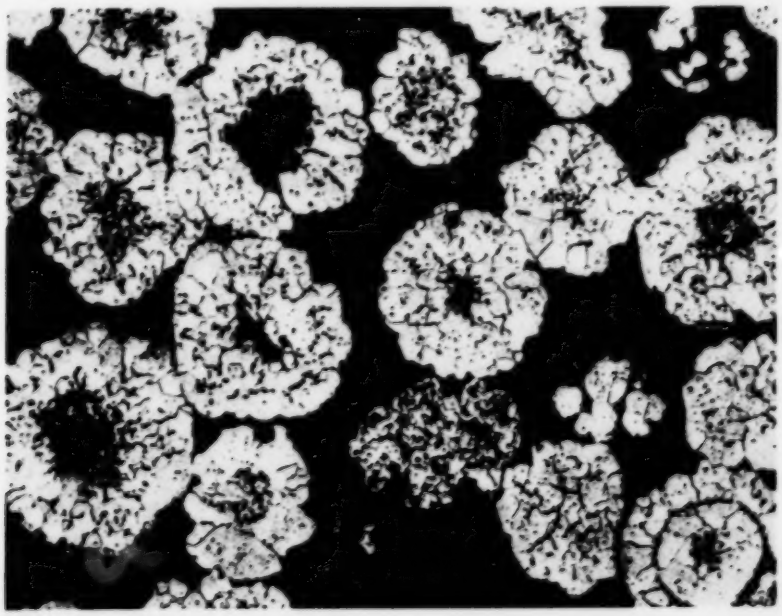


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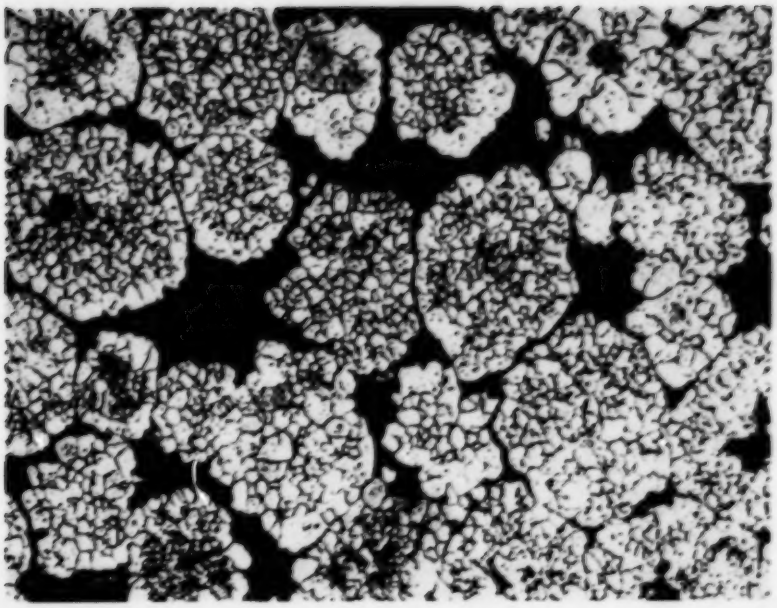
Figure 8. -100 +140S compacted at 42.7 ksi (295 MN/m^2) and sintered at 900°C (1650°F) for 2 hr.



Etched X200

Figure 9. -100 +140S compacted at 96 ksi (662 MN/m^2) and sintered at 900°C (1650°F) for 1/2 hr.

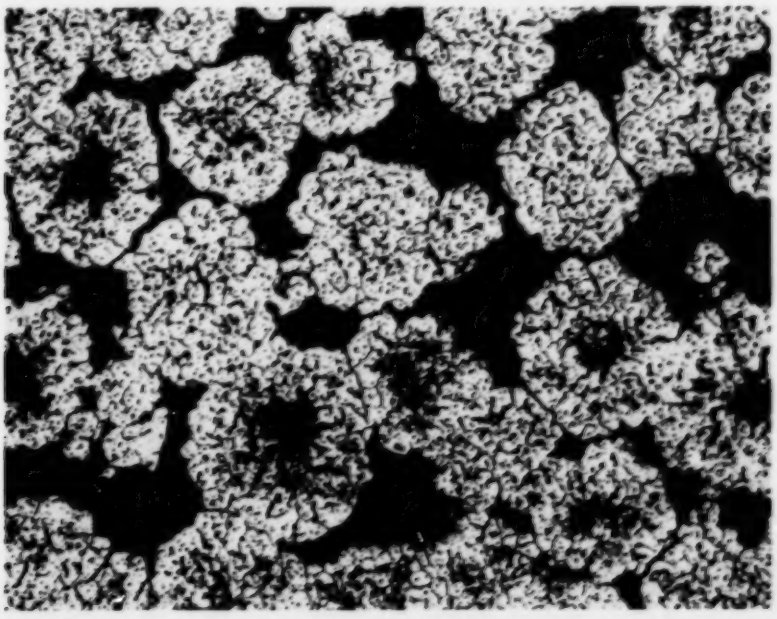


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Figure 10. -100 +140S compacted at 96 ksi (662 MN/m^2) and sintered at 900°C (1650°F) for 2 hr.

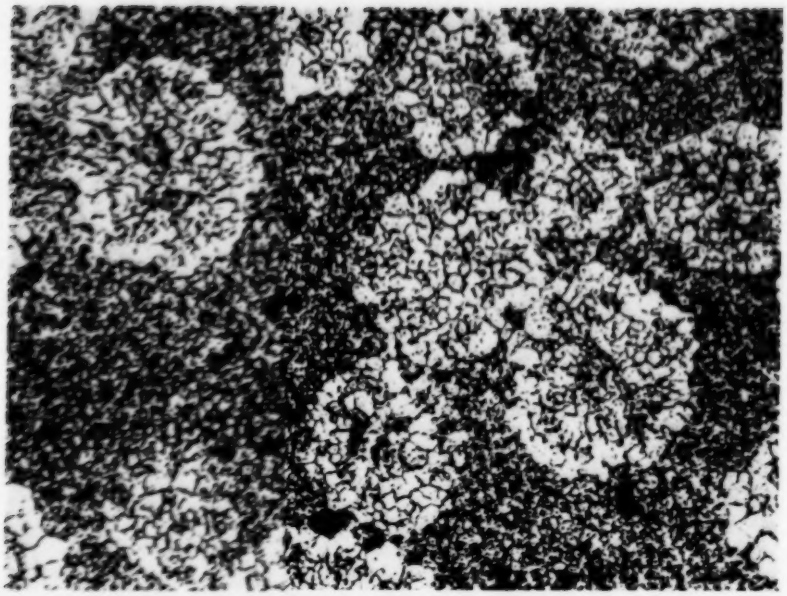

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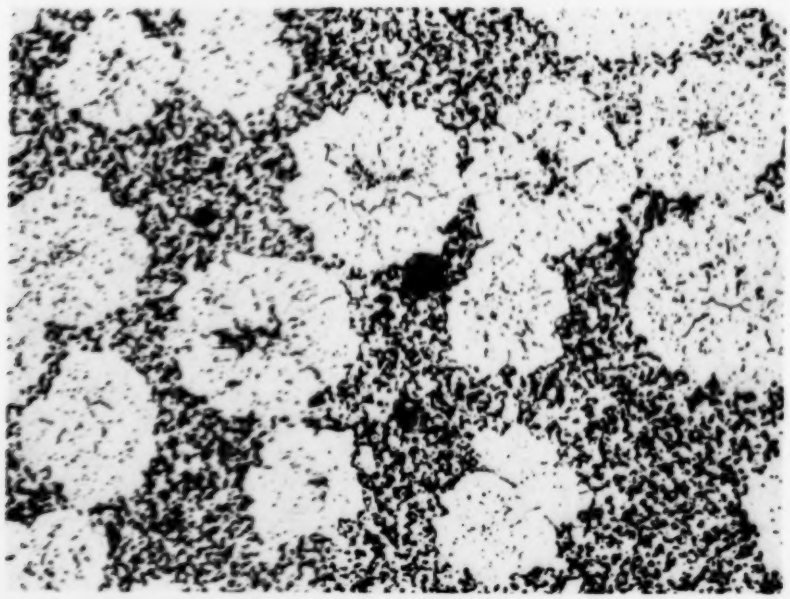
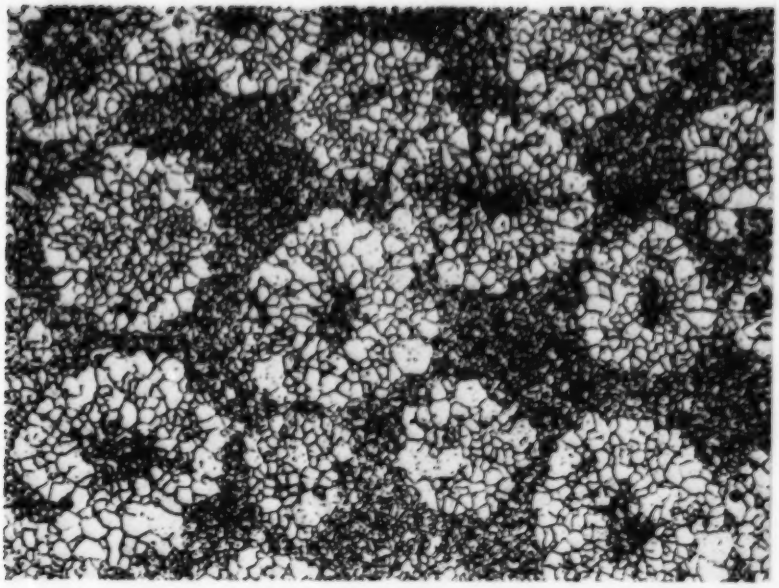
Figure 11. -100 +140S with 50% G-29 compacted at 42.7 ksi (295 MN/m^2) and sintered at 900°C (1650°F) for 1/2 hr.



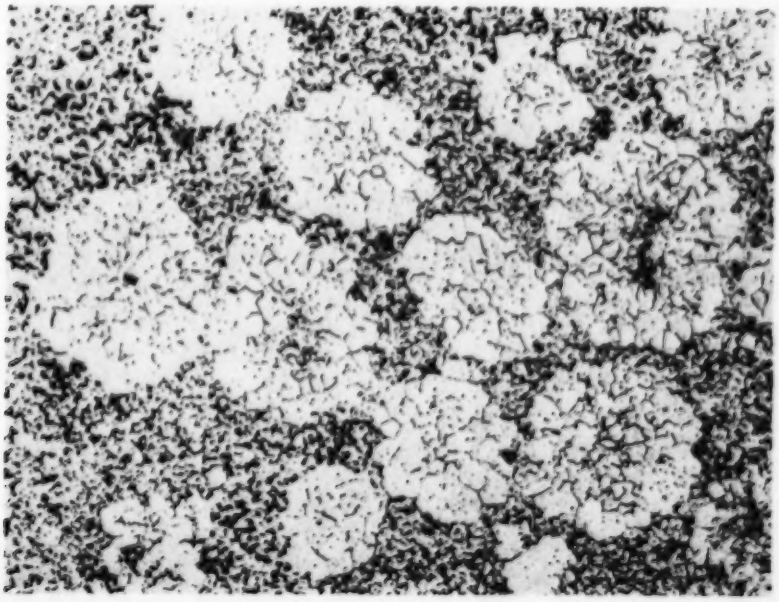
Etched X200

Figure 12. -100 +140S with 50% G-29 compacted at 42.7 ksi (295 MN/m^2) and sintered at 900°C (1650°F) for 2 hr.



Etched X200

Figure 13. -100 +140S with 50% G-29 compacted at 96 ksi (662 MN/m^2) and sintered at 900°C (1650°F) for 1/2 hr.



Etched X200

Figure 14. -100 +140S with 50% G-29 compacted at 96 ksi (662 MN/m^2) and sintered at 900°C (1650°F) for 2 hr.

(Etchant for Figures 7 to 14: Carapella's Reagent)